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PATENT ABSTRACTS OF JAPAN

(11)Publication number : 11-101943
(43)Date of publication of application : 13.04.1999

(51)Int.Cl. G02B 21/34
G01N 1/28
G01N 21/01

(21)Application number : 09-263022
(22)Date of filing : 29.09.1997

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(54) COVER FILM FOR MICROSCOPE

(57)Abstract:
PROBLEM TO BE SOLVED: To prevent blocking between an adhesive layer and the back face of a supporting body, and to prevent peeling of a film with time by incorporating a silane coupling agent into the surface of a polymer adhesive layer and/or into a polymer adhesive layer.
SOLUTION: This cover film for a microscope consists of a transparent supporting body and a polymer adhesive layer having ≥50°C glass transition temp. The surface of the polymer adhesive layer and/or the polymer adhesive layer contains a silane coupling agent. This cover film is produced by applying a silane coupling agent on the surface of a polymer adhesive layer having ≥50°C glass transition temp. applied on a transparent supporting body, or by applying a polymer adhesive layer containing a silane coupling agent. The silane coupling agent is an org. silicon monomer having two or more different reactive groups in the molecule, and one of the reactive group chemically bonds with an inorg. material, while the other reactive group chemically bonds with an org. material.

LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

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[Date of extinction of right]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] Peeling of this invention from slide glass according especially to aging about the covering film for microscopes suitable for automatic enclosure is small, and it is related with the covering film for microscopes suitable for automatic enclosure.
[Description of the Prior Art] The method of creating the sample for microscope observation is learned by what the covering film which applied and created adhesives on the transparent base material beforehand is automatically laid on top of the slide glass on which several drops of organic solvents which may swell or dissolve these adhesives were dropped, and the analyte was put, and is pasted up on it for (it is hereafter called enclosure). Using adhesives of 50 degrees C or more of glass transition temperatures for JP,5-19684,B for blocking prevention with the adhesives layer and film rear face (support surface of an adhesives layer and an opposite side) which are generated in such a covering film while saving in the state of a roll is indicated. However, when this film was pasted up on slide glass and it saved under the usual temperature-and-humidity environment for a long period of time, the film separated from slide glass and there was a problem of an analyte also separating from slide glass together, and damaging an analyte, depending on the case at this time. Therefore, a covering film which does not have peeling at the time of a mothball, either was desired.
[0002]
[Problem(s) to be Solved by the Invention] After it pastes up with cover glass and this invention encloses a sample, without blocking arising between an adhesives layer and a base material rear face even if saved by the shape of a roll, even if it saves it for a long period of time, it aims at offering the covering film for microscopes which peeling of the film by aging does not produce.
[0003]
[Means for Solving the Problem] In the covering film for microscopes with which a glass transition temperature comes to prepare a polymer adhesives layer 50 degrees C or more on a transparent base material, the above-mentioned covering film for microscopes characterized by making a silane coupling agent contain in the front face of this polymer adhesives layer and/or this polymer adhesives layer can attain the above-mentioned purpose of this invention.
[0004]
[Embodiments of the Invention] The covering film of this invention is created, when the glass transition temperature applied on the transparent base material applies a silane coupling agent further on the front face of a polymer adhesives layer 50 degrees C or more or applies the polymer adhesives layer containing a silane coupling agent on a transparent base material. After pasting up with cover glass and enclosing a sample, without blocking arising between an adhesives layer and a base material rear face even if saved by the shape of a roll by using such a covering film, even if it saves for a long period of time, peeling from the glass of a film stops arising. Here, a silane coupling agent has two or more different reaction machines in a molecule, one of the reaction machines is a reaction machine which carries out a chemical bond to minerals, and another is an organic silicon monomer which is the reaction machine which carries out a chemical bond to an organic material.
[0005] The silane coupling agent expressed with the following general formula as a desirable silane coupling agent of this invention is mentioned.
Y-R-Si(CH3) 3-n Xn (among a formula, Y expresses a vinyl, methacrylic one, epoxy, AMINO, a mercapto, or a crawl machine,;R expresses the polymethylene machine which single bond, a methylene group, a polymethylene machine, or at least one methylene group placed and replaced by O, S, or NH,;X expresses the crawl, a methoxy, ethoxy ** methoxyethoxy, acetoxy, methyl vinyloxy, or the amino group, and;n is 2 or 3.)

As a silane coupling agent used in this invention Vinyl trichlorosilan, a vinyl tris (beta-methoxyethoxy) silane, Vinyltriethoxysilane, vinyltrimetoxysilane, gamma-(methacryloxypropyl) trimethoxysilane, beta-(3, 4-epoxycyclohexyl) ethyl trimethoxysilane, Gamma-glycidoxypropyltrimetoxysilane, gamma-glycidoxypropylmethyldietoxysilane, N-beta-(aminoethyl)-gamma-aminopropyl trimethoxysilane, N-beta-(aminoethyl)-gamma-aminopropyl methyl dimethoxysilane, gamma-aminopropyl triethoxysilane, N-phenyl-gamma-aminopropyl trimethoxysilane, gamma-mercapto propyltrimethoxysilane and gamma-chloropropyltrimetoxysilane are desirable. Gamma-glycidoxypropyltrimetoxysilane, N-beta-(aminoethyl)-gamma-aminopropyl trimethoxysilane, and especially gamma-aminopropyl triethoxysilane are desirable.
[0006] It is easily understood by this contractor that the silane-coupling-agent kinds to be used are glass and the organic material which you want to paste up, i.e., the thing to select with an adhesives kind. Although there are a method which adds in adhesives beforehand, and applies to a base material and it is made to dry with adhesives as a method of making a covering film containing a silane coupling agent, and the method of applying a silane coupling agent on the front face of the adhesives layer which carried out application dryness on the base material, latter one is desirable for the ability of a silane coupling agent being harnessed a little and effectively. Moreover, there is also the method (multistory application) of applying adhesives and a silane coupling agent to a film simultaneously as the method of application. That is, two next persons have the lamination of a base material / adhesives / silane coupling agent. The content of the silane coupling agent in the covering film of this invention is 0.1 mg/m2. The above is desirable and they are 5 mg/m2 - 25 mg/m2. Between is the most desirable.
[0007] It is desirable to dissolve in the organic solvent used for automatic enclosure equipment as polymer adhesives used in this invention, and to swell with this solvent at least is required. It is desirable among a xylene, toluene, ethyl acetate, methyl acetate, an acetone, and a methyl ethyl ketone that it is fusibility to one of independent solvents, or two or more sorts of these mixed solvents. In case a covering film is saved in the shape of a roll, in order to prevent blocking with an adhesives layer and a base material rear face, it is meltable to the above-mentioned solvent, and the polymer adhesives whose glass transition temperature is 50 degrees C are desirable. As such polymer adhesives, Byron 200 (Toyobo Co., Ltd., Japan) is mentioned as polyester system adhesives, and Aron S-1017, Aron S-1030C (Toagosei Chemical industry, Japan), etc. are mentioned as acrylic adhesives.
[0008] It is possible to also compute the glass transition temperature of the copolymer which the formula of Fox is known (refer to Seizo Okamura work, the 2nd edition of a polymer-chemistry introduction, and Kagaku-Dojin (172 pages)), and consists of three or more sorts of monomers by the same method about calculation of the glass transition temperature of the copolymer which consists of two sorts of monomers. The calculation method is indicated in detail by JP,5-19684,B, and the whole of this content is quoted here. A thing desirable as acrylic adhesives which consist of two sorts of monomers is the copolymer of alkyl methacrylate and alkyl acrylate. The refractive index of the coat of these polymer adhesives (etaD) From the optical problem in the case of microscope observation, it is desirable that it is 1.45 or more, and it is still more desirable that it is 1.47 or more.
[0009] Although these polymer adhesives may be used independently, they may blend and use two or more sorts of polymer adhesives. When blending and using it, it is necessary to use for the coat after the organic solvent evaporates by the ratio which muddiness does not produce. The coverage of the polymer adhesives layer after the organic solvent evaporates is about 1 g/m2 - 50 g/m2. Between is desirable and they are 7 g/m2 - 25 g/m2. Between is the most desirable. It is desirable that a glass transition temperature blends and uses polymer adhesives (for example, acrylic polymer adhesives) 80 degrees C or more and polymer adhesives (for example, acrylic polymer adhesives) with a glass transition temperature smaller than 50 degrees C or more 80 degrees C in the range of the weight ratio of 7:3-5:5 as a mode which blends the aforementioned polymer adhesives.
[0010] Moreover, in order to prevent more certainly the blocking at the time of a crack being sufficient for a silane-coupling-agent front face, a polymer adhesives front face, and/or a support surface just, and being saved very much in an elevated temperature at them, or in order to maintain curling balance again, a protective layer may be painted on this silane coupling agent and/or an adhesives layer, or a backing layer may be painted on the rear-face side (adhesives and opposite side) of a base material. As a constituent of a protective layer and a backing layer, the synthetic high polymer of glass transition temperatures, such as polystyrene and a polymethylmethacrylate, or gelatin is mentioned, for example.
[0011] Although all the things known in the world as a transparent base material can be used as a base material of this invention, as a desirable example, a triacetate cellulose wall, a diacetate cellulose wall, and a polyethylene-terephthalate film are mentioned. As for the thickness of these base materials, for 50 micrometers - 250 micrometers is desirable, and while being 100 micrometers - 150 micrometers is the most desirable.

Lower ****** well known for the photosensitive-material industry may be painted on these base materials, or surface treatment, such as UV irradiation, corona discharge, or glow discharge, may be made.
[0012]
[Example]
A triacetate cellulose with one to example 4 base material transparent [the thickness of 120 micrometers] and polymer adhesives used what blended Aron S-1017 and Aron S-1030C (all are Toagosei Chemical industry and Japan) by the weight ratio 2:3. The glass transition temperature of these polymer adhesives was computed with about 65 degrees C from the formula of Fox. A silane coupling agent uses KBM403 (gamma-glycidoxypropyltrimetoxysilane) and KBM602 (N-beta-(aminoethyl)-gamma-aminopropyl methyl dimethoxysilane) (Shin-Etsu Chemical Co., Ltd.), respectively, and is 5 mg/m2 - 100 mg/m2. It applied to the polymer adhesives layer front face, and the covering film shown in Table 1 was created. The application method used the extrusion multistory application method. Polymer adhesives and the silane coupling agent were diluted with ethyl acetate, respectively, and were applied, respectively. Polymer adhesives (ARON blend) performed dilution concentration, and performed the silane coupling agent at 0.1% 55%. Dryness was performed for 5 minutes at 100 degrees C after the application. The thickness after dryness of adhesives was 19 micrometers. These covering films were stuck with slide glass using covering aid automatic enclosure equipment SCA-1800 (Cherry Energy Machine, Japan). Each covering film was put after enclosure on the bottom of 40 degrees C and 80% (RH) of environment, and the film peeling accelerated test was performed.
[0013] Only polymer adhesives were applied to the base material like example of comparison 1 examples 1-4, it dried and the covering film was created. It stuck with slide glass after the application, and the film peeling accelerated test was performed like examples 1-4.
[0014]
[Table 1]
Silane coupling agent Example Kind Addition Film peeling situation 1 KBM403 5 mg/m2 With no peeling (it evaluates to a 200 Nikkei fault)
2 KBM403 50 Mg/m2 Same as the above 3 KBM403 100 Mg/m2 Same as the above 4 KBM602 5 Mg/m2 With No Peeling (it Evaluates to 100 Nikkei Fault)
Example of comparison 1 Nothing - It will begin to separate in progress for 20 days. It has separated altogether in progress for 90 days.
[0015] Although peeling of a film did not arise for a long period of time when the covering film which applied the silane coupling agent was used so that clearly from Table 1, when the covering film which does not apply a silane coupling agent is used, it has separated altogether within 90 days.
[0016]
[Effect of the Invention] Although that to which the glass transition temperature laid on top of slide glass with the covering film for microscopes only using adhesives 50 degrees C or more, and carried out enclosure adhesion of the sample had the problem that a film will separate from slide glass when it was saved under the usual temperature-and-humidity environment for a long period of time, it stopped producing ablation of the covering film by aging by making a silane coupling agent contain in an adhesives layer front face and/or an adhesives layer.

[Translation done.]

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
011515wo Me/tg		
International application No.	International filing date (day/month/year)	Priority date (day/month/year)
PCT/US 01/ 00996	11/01/2001	07/08/2000
International Patent Classification (IPC) or national classification and IPC	EPO - DG 1	
G01N1/36		
Applicant	13. 09. 2002	
3M INNOVATIVE PROPERTIES COMPANY		

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1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 2 sheets, including this cover sheet.

☐ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consists of a total of _____ sheets.

3. This report contains indications relating to the following items:

I ☒ Basis of the report

II ☐ Priority

III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

IV ☐ Lack of unity of invention

V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

VI ☐ Certain documents cited

VII ☐ Certain defects in the international application

VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
03/07/2001	12/06/2002
Name and mailing address of the IPEA/	Authorized officer
European Patent Office D-80298 Munich Tel. (+49-89) 2399-0, Tx: 523656 epmu d Fax: (+49-89) 2399-4465	STOOS A Tel. (+49-89) 2399 2828

Form PCT/IPEA/409 (cover sheet) (July 1998)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No PCT/ US 01/ 00996

I. Basis of the report

The basis of this international preliminary examination is the application as originally filed.

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability

In light of the documents cited in the international search report, it is considered that the invention as defined in at least some of the claims does not appear to meet the criteria mentioned in Article 33(1) PCT, i.e. does not appear to be novel and/or to involve an inventive step (see international search report, in particular the documents cited X and/or Y and corresponding claim references).

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 01/00996

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 G01N1/36 G02B21/34 C09J7/02 C09J133/08 C09J133/10

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 G01N G02B C09J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 853 262 A (HORIE IKUTARO ET AL) 1 August 1989 (1989-08-01) column 4, lines 19-21 column 3, line 57 to column 4, line 3 claims 1-3	1-3,8-20
A	WO 99 53357 A (3M INNOVATIVE PROPERTIES CO ;GOVEK MICHAEL (US); CHAFFEE LINDA C () 21 October 1999 (1999-10-21) claim 1	1-20
A	US 3 498 860 A (PICKETT JOHN E P) 3 March 1970 (1970-03-03) the whole document	1-20

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[X] Further documents are listed in the continuation of box C. [X] Patent family members are listed in annex.

° Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier document but published on or after the international filing date
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"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
4 May 2001	16/05/2001

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Rousseau, F

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 01/00996

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DATABASE WPI Section PQ, Week 199925 Derwent Publications Ltd., London, GB; Class P81, AN 1999-297906 XP002166495 & JP 11 101943 A (FUJI PHOTO FILM CO LTD), 13 April 1999 (1999-04-13) abstract	1-20

Form PCT/ISA/210 (continuation of second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/US 01/00996

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4853262 A	01-08-1989	JP 1814392 C	18-01-1994
		JP 5019684 B	17-03-1993
		JP 62038408 A	19-02-1987
WO 9953357 A	21-10-1999	AU 3744299 A	01-11-1999
		EP 1070273 A	24-01-2001
US 3498860 A	03-03-1970	NONE	
JP 11101943 A	13-04-1999	NONE	

Form PCT/ISA/210 (patent family annex) (July 1992)